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Treatment of sugar decolorizing resin regeneration waste using nanofiltration

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Abstract

In the sugar industry, ion-exchange resins have a problem with regeneration waste disposal since the regeneration effluent contains a high concentration of sodium chloride and colored organics. In the present work the recovery and recycling of waste brine previously used for decolorizing sugar liquor has been investigated using cost-effective organic spiral-wound nanofiltration membranes. A 74% reduction in salt consumption and an 89% reduction in water consumption were achieved in the batch configuration at VCF 9.

Keywords: Nanofiltration; Ion exchange resins; Regeneration effluent

1. Introduction

In the sugar industry, ion exchange-resins are mainly used to remove colorants from cane sugar liquor. The regeneration of the resins is carried out by passing an alkaline brine solution through the resin to desorb colorants. The resulting resin regeneration waste, characterized by high salinity and chemical oxygen demand (COD), is particularly polluting, which raises the problem of disposal. In the present work, a nanofiltration (NF) process has been investigated for the treatment of resin regeneration effluent to (1) recover spent brine for reuse and (2) reduce the discharge of pollutant waste. The primary goal of this study was to select the proper fraction of the effluent that could be efficiently treated by NF membranes. The secondary goal was to experimentally evaluate NF membrane performances in terms of productivity and quality of the recovered brine, and effluent volume reduction by varying operating conditions.

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2. Background

2.1. Nanofiltration process

Nanofiltration is a relatively new pressure-driven membrane filtration process, falling between reverse osmosis and ultrafiltration, which has significantly widened the application of membranes in liquid-phase separations [1]. Presently, most of the commercially available NF membranes are negatively charged. While large inorganic and organic molecules with a molecular weight greater than 300 Daltons are retained by NF membranes because of their size, ionic species are selectively retained according to their charge densities. This means that monovalent ions in salts such as sodium chloride tend to permeate NF membranes, and salts containing multivalent anions such as sodium sulphate are highly retained. Actually, the NF mechanism is based on the concept of molecular exclusion as well as on the principle of electrostatic repulsion.

2.2. Previous work on sugar decolorizing resin regeneration waste treatment

The ion-exchange resin process is currently considered as one of the most efficient sugar liquor decolorizers [2]. High-molecular weight sugar liquor colorants such as melanines, melanoidines, products of alkaline degradation of sucrose, caramels, and polyphenols are first adsorbed onto the resins, and finally desorbed from the exhausted resins using an alkaline 100 g/l sodium chloride solution at approximately pH 12. Effluents resulting from this regeneration contain mostly sodium chloride (up to 100 g/l) and important amounts of colored organic matter, and, therefore, constitute a major pollution source.

New environmental regulations placed under strong constraints have forced the sugar industry to develop economical innovative approaches to reduce the amount of waste solutions for disposal. In this context, different methods for spent brine waste reduction were investigated on a laboratory scale:

1. Oxidations of color bodies to carbon dioxide by calcium hypochlorite, as well as chlorine gas were found to be inefficient and extremely expensive [3].

2. Ozone was also used to oxidize colored compounds. However, incomplete oxidation and subsequent recombination of broken down organic matter into colorants prevented further study [4].

3. While carrying out a first regeneration at low sodium chloride concentration and a second one at normal sodium chloride concentration, Bento managed to differentiate two types of colorants according to their anionic affinities to the resins. Only the effluent resulting from the second regeneration could be precipitated with lime. After filtration, the solution containing mostly non-anionic colorants was satisfactorily reused to regenerate the loaded resins [4,5]. Although a promising 60% reduction in effluent volume was reported, this process cannot be easily integrated into an ion-exchange plant because of space requirements and need for expensive lime sludge equipment.

4. The potential of crossflow filtration to treat regeneration effluent was investigated by several workers [6–8]. According to Wilson and Percival, ultrafiltration induced a 45% reduction in organic matter in the portion recycled as regenerant, which represents 40% of the whole effluent [6]. This low organics retention suggests that ultra-fitration membranes are not tight enough. Furthermore, the productivity aspect had not been approached by these authors.

Tubular organic NF membranes were used to treat the salt-rich fraction of the regeneration effluent [7,8]. Such tubular configuration allows operation at high temperatures (<70°C) and pH (<12) [9]. Using the SelROTM MPT-30 and MPT-31 membranes at 45°C and 60°C and an operating pressure of 30 bar, the experimental work was focused on a portion including 86% of the sodium chloride and 37% of the organic compounds contained in the effluent, corresponding to a pH between 8 and 9. The NF

process was recognized as technically feasible on a pilot plant scale and demonstrated a 30% reduction in effluent volume and a 60% reduction in salt consumption at Huletts Refineries.

3. Scope of the project

The purpose of the present work was to investigate the feasibility of colorant removal from resin regeneration effluent using organic spiral-wound NF membranes. This configuration is largely applied with streams characterized by both low viscosities and suspended solids [10]. Despite the alkalinity of this effluent, the spiral-wound type was selected as its operating costs are generally three times lower than those of the tubular configuration [9]. Since regeneration waste contains mostly sodium chloride and high molecular-weight species negatively charged at alkaline pH, NF seems to be the most appropriate separation process to recover good-quality brine in the permeate and to concentrate organic matter in the retentate. Specifically, the NF process aims at splitting the waste stream into a colorless diluted stream containing monovalent ions that can be recycled and a highly colored concentrated stream containing most of the pollutant compounds, as illustrated in Fig. 1. Thus, the quantity of waste product can be disposed of more easily at less cost since its volume is only a fraction of the original spent brine. Moreover, the recovery of useable brine from spent brine should provide potential savings on both salt and water consumptions for ion exchange regeneration process.

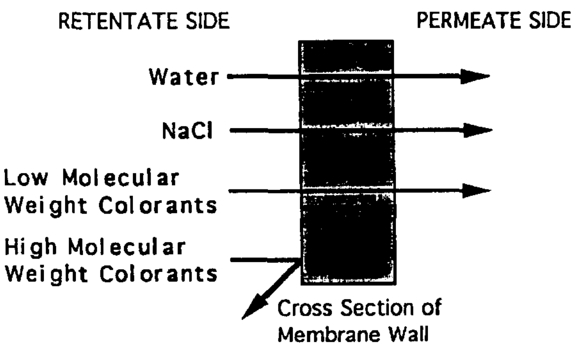

Fig. 1. Schematic representation of the nanofiltration process applied to brine recovery and colorants concentration.

4. Materials and methods

4.1. Pilot plant equipment

On-site pilot-plant tests were conducted using a 2.5" diameter by 40" long module on a pilot plant purchased from SLCE (Caudan, 56850 France). The experimental set up is shown in Fig. 2. The retentate flow rate and pressure could be varied by manually operating valve V1 that was downstream of the membrane. The permeate and retentate flows could be monitered by flow meters installed respectively in the permeate and retentate lines.

The ideal candidate membrane should exhibit a high rejection of organic components and a low rejection of monovalent ions. Based on technical

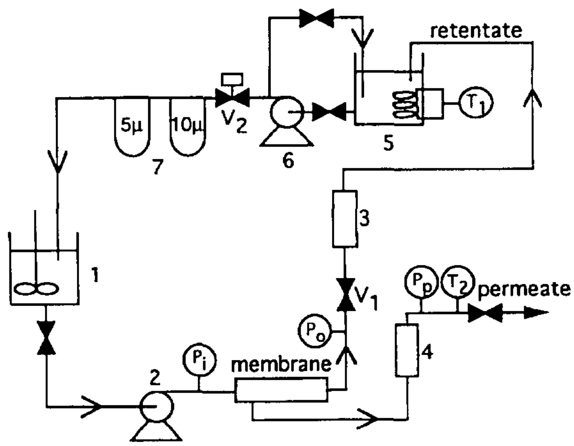

Fig. 2. Schematic experimental apparatus for NF. 1 50-l feed tank; 2 volumetric circulation pump; 3 retentate flow meter; 4 permeate flow meter; 5 2000-l feed tank with heating system; 6 feed pump; 7 cartridge filters; P_i inlet pressure (bar); P_o outlet pressure (bar); P_p permeate pressure (bar); T_1 2000-l feed tank temperature (°C); T_2 permeate temperature (°C); V_1 pressure valve; V_2 electrovalve.

information given by membrane manufacturers, we selected the organic spiral-wound Desal 5 membrane supplied by Desalination Systems Inc. (CA, USA) which shows a high retention for organics. Its molecular weight cut-off ranges from 150 to 300 Daltons. The spiral wound configuration is cost-effective since it allows a large volume of effluent to be passed through the membrane. Two samples of identical membranes referred to as Membrane Desal 5.1 and Membrane Desal 5.2 were used in order to eliminate the possible effects of membrane nonhomogeneity caused during the manufacturing process. A one-batch concentration run was conducted with the organic spiral-wound Filmtec NF45 membrane (Dow Europe Separations Systems, Germany) to compare membranes performances. The NF membranes tested were made from a variety of polymeric materials such as polysulfone and polyamide. The manufacturers recommended an operating pH range between 2 and 12 and an operating temperature below 40°C. The membrane area was approximately 2.1 m^2.

The regeneration effluent was provided by a sugar refinery. The effluent composition will be thoroughly studied later.

4.2. Experimental method

Prior to NF, the effluent was filtered through a 10 and 5 μ cartridge safety filter to eliminate any potential suspended solids that could damage the membranes.

Applied pressure across the membranes was varied in the range of 10 to 30 bars. THe temperature of the feed solution was kept at 30°C. Because of membrane limited pH-resistance, the pH of the feed solutions was lowered from 13 to 11 using hydrochloric acid.

The on-site pilot plant was first operated in a total recycle mode, which means that both permeate and retentate were returned to the feed tank during 60 min. Batch concentrations were then performed for an average period of 6 h by continuously extracting permeate and recycling retentate until equipment constraints prevented further permeate recovery. Permeate flow rates, pressure and permeate volume were monitered throughout the experiment.

After completion of the experiment, membrane cleaning was carried out by first rinsing with water, then circulating a 0.02% alkaline solution for 60 min. When more than 2 d had elapsed between runs, membranes were stored in a 0.1% solution of sodium bisulphite to inhibit microbiological activity.

The performance characteristics of the membranes before and after sodium hydroxide cleaning were periodically examined by determining permeate flux and sodium chloride retention at a given sodium chloride concentration.

4.3. Analytical method

Permeate and retentate samples were collected for conductivity analyses and optical density measurements during the operation of the NF system at regular time intervals in order to study species-rejection characteristics.

Optical density (OD) was chosen as a measure for the colorant concentration in the chemically complex industrial effluent. The similar evolution of OD and COD during a typical regeneration cycle shown in Fig. 3 confirms that colored matter is the main polluting organic species. Organic pollution abatement was therefore characterized in terms of color removal. The OD was measured at 420 nm using a Perkin-Elmer spectrophotometer.

Chlorides and COD analyses were performed by the Institut de Recherches de l'Industrie Sucrière (IRIS) according to their standard protocol.

5. Definitions

Membrane processes are generally evaluated in terms of three parameters: permeate flux (J_p), solute retention (Ret_{NaCl} and Ret_{Col}) which is

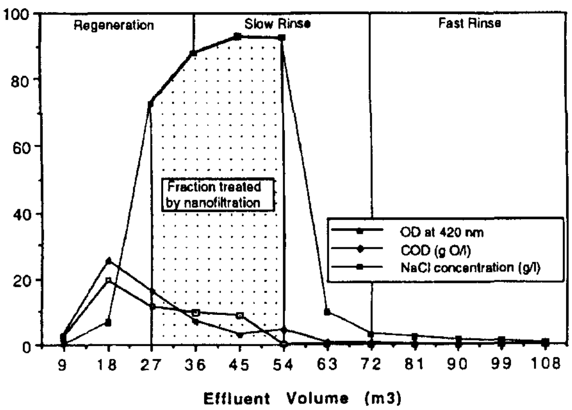

Fig. 3. Regeneration profile as a function of effluent volume for sodium chloride concentration, chemical oxygen demand and optical density value. (One bed volume corresponds to 14 m^3).

determined by using instantaneous permeate and retentate concentrations or OD, and the volumetric concentration factor (VCF).

$$\mathrm{Ret}_{\mathrm{NaCl}}\,(\%) = \left(1 - \frac{[\mathrm{NaCl}]_p}{[\mathrm{NaCl}]_r}\right) \times 100$$

and

$$\mathrm{Ret}_{\mathrm{Col}}\,(\%) = \left(1 - \frac{\mathrm{OD}_p}{\mathrm{OD}_r}\right) \times 100$$

$$\mathrm{VCF} = \frac{V_f}{V_r}$$

where V represents the volumes, and subscripts f, p, and r stand, respectively, for feed, permeate, and retentate.

The membrane performances can also be characterized by their capacity for recovering sodium chloride ($\mathrm{Rec}_{\mathrm{NaCl}}$), and removing colorants ($\mathrm{Rem}_{\mathrm{Col}}$) in the permeate. Both terms derive from mass balance considerations:

$$\mathrm{Rec}_{\mathrm{NaCl}}\,(\%) = \frac{[\mathrm{NaCl}]_p \times V_p}{[\mathrm{NaCl}]_f \times V_f} \times 100$$

$$= \frac{[\mathrm{NaCl}]_p}{[\mathrm{NaCl}]_f} \times \left(1 - \frac{1}{\mathrm{VCF}}\right) \times 100$$

$$\mathrm{Rem}_{\mathrm{Col}}\,(\%) = \left(1 - \frac{\mathrm{OD} \times V_p}{\mathrm{OD}_f \times V_f}\right) \times 100$$

$$= \left[1 - \frac{\mathrm{OD}_p}{\mathrm{OD}_f} \times \left(1 - \frac{1}{\mathrm{VCF}}\right)\right] \times 100$$

6. Results and discussion

6.1. Characterization of the regeneration effluent

The nature of the regeneration effluent was preliminary investigated to select the fraction that was the most suitable to treatment by NF. Effluent samples were taken at 9 m^3 intervals during a regeneration cycle. It has to be specified that the resin vessel contains 14 m^3 of resin. Fig. 3 displays the evolution of the composition of the effluent that had passed through the resin vessel in terms of sodium chloride concentration, COD and OD. These curves showed that initially COD and OD increase much faster than sodium chloride concentration. The fraction situated between 27 m^3 and 54 m^3 contains 93% of the total sodium chloride and 50% of the total COD present in the effluent. In order to recover a salt permeate concentration high enough for recycling, the NF work focused on this effluent portion which represents 36 m^3 per regeneration or 2.6 bed volumes (BV) of resin per regeneration (1 BV=14 m^3).

As a result, a 600 l volume of composite effluent taken at 9 m^3 intervals from 27 m^3 to 54 m^3 during the regeneration cycle, and, therefore representative of the average

composition of the treated effluent, was processed for all runs.

6.2. Effect of time on flux performance (total recycle mode)

Total recycle mode experiment was made to determine the typical effect of time on the permeate flux. Membrane Desal 5.2 was operated at 15 bars over a 4-h period. The plot of flux vs time is shown in Fig. 4. A sharp flux decline from 72 l/h/m^2 to 55 l/h/m^2 was observed during the first 3 h. After this period the permeate flux remained nearly constant. This could be due to either fouling of the membrane or interaction between effluent and membrane which has been extensively reported in literature [11]. The initial water flux was 91 l/h/m^2 whereas the water flux after the total recycle run was 70 l/h/m^2. The value of the initial water flux was subsequently restored after cleaning with sodium hydroxide, as demonstrated in Fig. 4. This indicates that flux decline results mainly from membrane fouling. The duration of the total recycle run is sufficient to estimate the time effect on short-term runs. However, the time effect should also be extensively studied by carrying out longer-term experiments.

The membrane selectivity, sodium chloride and colorant retentions were, respectively, estimated at 3.7% and 96.4% at the beginning of the experiment, and at 4.1% and 97.1% after 270 min. Therefore, these consistent results are in agreement with the expected retention of the membrane used which retains large organic molecules and allows the passage of monovalent ions.

6.3. Flux performances in batch concentration runs

Batch concentration runs were conducted to assess the effect of colorant concentration on permeate flux. Operating conditions, permeate flux, and the volumetric concentration factor corresponding to each batch run are listed in Table 1. Operating conditions were set according to the manufacturer's recommendations. It should be emphasized that initial optical densities of feed vary from 4.7 to 30.3. The large disparity in the industrial effluent composition is mainly caused by variations of effluent sampling operation. In addition, influent sugar color, service run length and regenerant dosage may as well induce fluctuations in industrial effluent composition. However, these on-site experiments enabled to cover a wide colorants concentration range.

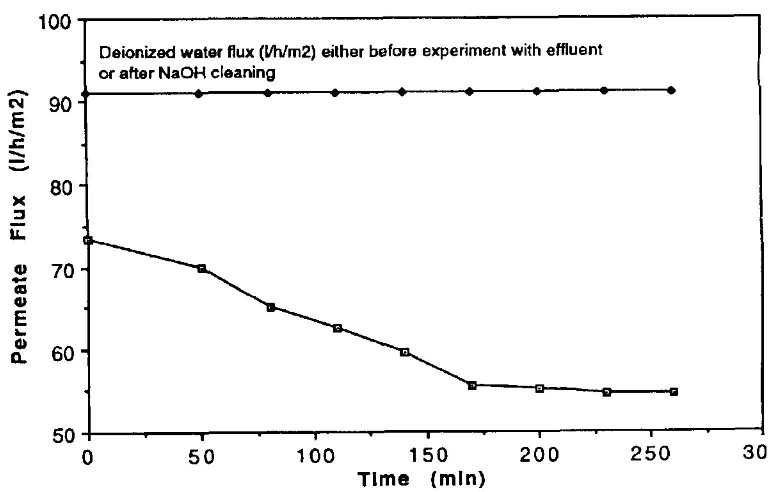

Fig. 4. Effect of time on permeate flux for total recycle experiment at 15 bar and 30°C with Membrane Desal 5.2. Initial feed: [NaCl] = 71.1 g/l and OD = 10.6; pH = 11. At t=0: Ret_{NaCl} = 3.7% and Ret_{Col} = 96.4%. At t = 250 min: Ret_{NaCl} = 3.9% and Ret_{Col} = 97.1%.

Table 1
Operating conditions and membrane flux performances in different batch experiments

Run	Membrane type	VCF	P, bar	J_p, l/h/m^2		OD of retentate at 420 nm	
				Initial	Final	Initial	Final
1	Desal 5.1	3.3	10	95	47	4.7	11.4
2	Filmtec NF45	6.2	25	50	13	10.4	71.0
3	Desal 5.2	6.5	20	62	32	11.1	67.5
4	Desal 5.2	2.9	20	50	25	30.3	75.0
5	Desal 5.2	8.1	30	55	30	10.7	49.8
6	Desal 5.2	8.9	30	62	30	19.1	116.0
7	Desal 5.2	3.0	20	55	36	10.6	28.9
8 [a]	Desal 5.1	4.9	10–20	62	50	16.2	55.9

[a]Run 8 was performed by increasing the pressure at a rate of 2 bar per hour.

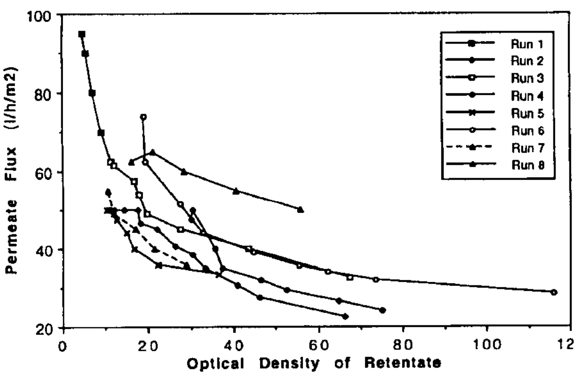

Fig. 5. Evolution of permeate flux with increasing OD of retentate during batch concentration experiments with different membranes at various operating conditions, as listed in Table 1.

A plot of permeate flux vs OD of retentate is represented in Fig. 5. Despite the variations of the feed composition, the curves present overall a similar trend: permeate flux decreases as OD of retentate increases, and therefore, as concentration of colorants increases. From this graph, it is reasonable to deduce that permeate flux is strongly affected by colorant concentration. On one hand, initial permeate flux is lower at higher colorant feed concentration, as especially noticed in Run 4. On the other hand, the higher the colorant concentration is in the retentate, the higher the osmotic pressure becomes in the retentate which decreases the effective pressure across the membrane, hence inducing a flux decline. Furthermore, the increasing colorant concentration in the retentate may extent membrane fouling through membrane/retained solute interactions.

Membrane Desal 5.1 in Run 1 demonstrates an initial permeate flux much higher than that given by Membrane Desal 5.2 in Run 7. This is partially related to the lower initial OD in Run 1. However, the flux through Membrane 1 drops drastically, and finally, product rates for both membranes present the same magnitude, as seen in Run 8 and Run 3. This result indicates that both these two membrane samples are not exactly identical.

The membrane NF45 used in Run 2 exhibits a similar permeate flux vs the OD curve to those obtained with the membranes Desal 5.

6.4. *Effect of pressure on permeate flux*

Applied pressure adjusted at 20, 25 and 30 bars did not seem to be a relevant factor for flux performance. The flux profile could be only slightly improved by regularly increasing the pressure at a rate of 2 bars per hour to prevent the permeate flux through Membrane Desal 5.1 from declining (Run 8). Although the pressure increase

did initially overcome fouling, a flux decline still occured as the OD increased. Considering that operating pressure is a good index of operating and capital costs, it turns out that it is not worth working at too high pressures, especially since the NF process is not particularly time-limited.

6.5. Solute retention

The typical dependences of colorants and NaCl retention on the volumic concentration factor are respectively illustrated in Figs. 6 and 7. The typical retention ranging from 98.6% to 99.6% in Run 3 proves that colorants are greatly retained by the membrane, assuring the production of good-quality permeate brine. It is also seen that colorant retention increases as concentration of the feed solution proceeds. This can be attributed to the fact that the average molecular weight of species remaining in the retentate increases during a batch experiment. In addition, the build-up of the highly charged layer of colorants on the membrane surface may also increase the rejection of those colorants at high VCF. By contrast, the retention of sodium chloride is particularly low since it drops from 4% to −4%. In addition, the sodium chloride retention decreases gradually as VCF increases and becomes even negative from a certain VCF value, resulting in maximal salt recovery in the permeate. This phenomenon, known as the Donnan effect, is ascribed to the enhancement of the passage of chloride ions through the membrane caused by the increasing concentration of retained charged organics in the retentate [12]. Due to this low salt retention, only a slight accumulation of chloride takes place in the circulation loop. Consequently, chloride ions are negligibly involved in the osmotic pressure increase. This constitutes the main advantage of using NF rather than reverse osmosis which requires a much higher operating pressure to overcome the osmotic pressure.

6.6. Salt recovery and colorant removal in the permeate

Table 2 summarizes the calculated colorant removal and salt recovery in the permeate obtained for each batch experiment. Uniformly high colorant removal varying from 97.1% to 99.3% is achieved whereas 66.6% to 90.4% of the salt is recovered, depending upon the final VCF value. As expected, the salt recovery in the permeate increases with increasing VCF. Therefore, one needs to find a compromise

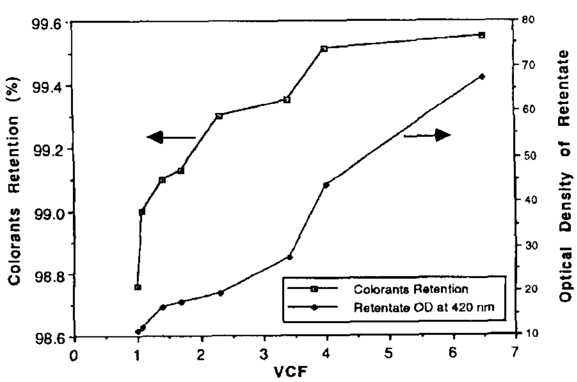

Fig. 6. Retention of colorants as a function of volumic concentration factor during Run 3 at 20 bar and 30°C with Membrane Desal 5.2. Initial feed: [NaCl] = 78.4 g/l and OD = 11.1; pH = 11.

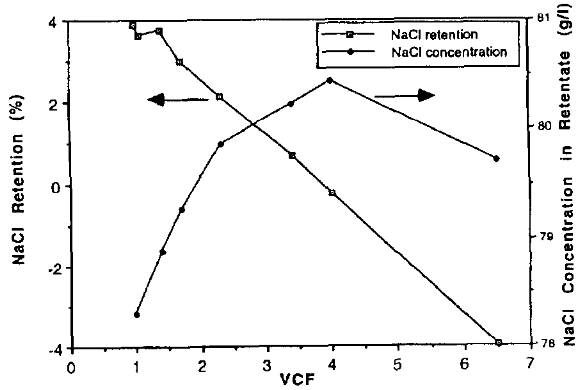

Fig. 7. Retention of sodium chloride in the retentate as a function of volumic concentration factor during Run 3 at 20 bar and 30°C with Membrane Desal 5.2. Initial feed: [NaCl] = 78.4 g/l and OD = 11.1; pH = 11.

Table 2
Colorant removal and sodium chloride recovery in the permeate during batch experiments

Run	VCF	OD at 420 nm			Colorant removal in permeate, %	NaCl concentration			NaCl recovery in permeate, %
		Feed	Permeate	Retentate		Feed	Permeate	Retentate	
1	3.3	4.7	0.16	11.4	97.6	49.0	46.8	48.8	66.6
2	6.2	10.4	0.12	71.0	99.0	66.8	64.5	72.3	81.0
3	6.5	11.1	0.18	67.5	98.6	78.4	78.0	79.7	84.2
4	2.9	30.3	0.32	75.0	99.3	62.9	64.0	64.6	66.7
5	8.1	10.7	0.17	49.8	98.6	73.7	73.2	77.8	87.0
6	8.9	19.1	0.53	116.0	97.5	81.8	83.3	83.1	90.4
7	3.0	10.6	0.45	28.9	97.1	71.1	71.4	72.9	67.0
8	4.9	16.2	0.44	55.9	97.8	76.9	81.6	81.1	84.4

between product rate which decreases as VCF increases, and, recovery. These results demonstrate that, in Run 6 for instance, the NF process allows production from regeneration effluent waste, 89% of brine permeate at a 83.3 g/l sodium chloride concentration while eliminating 97.5% of the colorants in the permeate. Subsequent steps would be to add sodium chloride and sodium hydroxide to the recovered brine to restore the regenerant components to their original concentration and pH. As the conventional regeneration process is carried out using 36 m^3 of brine at about 100 g/l sodium chloride concentration, the recovery of permeate by NF results in the respective 74% and 89% reductions on initial salt and water consumptions per regeneration cycle in the case of Run 6. At the same time, 50% of the pollutant species generated during regeneration are concentrated in 11% of the volume that would be discharged with no treatment. This reduction in effluent volume would facilitate secondary treatment of the regeneration effluent.

The results reported here can be compared with those reported in the study on NF of the salt-rich fraction conducted by Meadows et al. [7]. A higher reduction on salt consumption was achieved in this work (74% against 60% in Meadow's study) since the effluent feed that contained 93% of the sodium chloride had been concentrated until the volumic concentration factor reached 9 (in Meadow's study the effluent containing 86% of the sodium chloride was concentrated up to VCF 4).

6.7. *Effect of regeneration effluent on the performances of the membranes tested*

Figs. 8 and 9 illustrate the influence of regeneration effluent contact on membrane performances. Permeate flux and sodium chloride retention were measured with a 20 g/l sodium chloride concentration at 15 bars to characterize initial membrane performances in terms of product rate and salt retention. The same test was repeated after batch concentration runs that were immediately followed by cleaning in sodium hydroxide for 1 h. No major modification was detected regarding permeate flux and sodium chloride retention apart from Membrane Desal 5.1, indicating that membrane exposure to effluent does not entail membrane deterioration during batch experiments over a 6-h period.

Membrane Desal 5.1 exhibited both lower permeate flux and sodium chloride retention after Run 1. Subsequent sodium hydroxide cleaning was not sufficient to restore the initial permeate flux value. This implies that Membrane Desal 5.1 was irreversibly modified after one batch

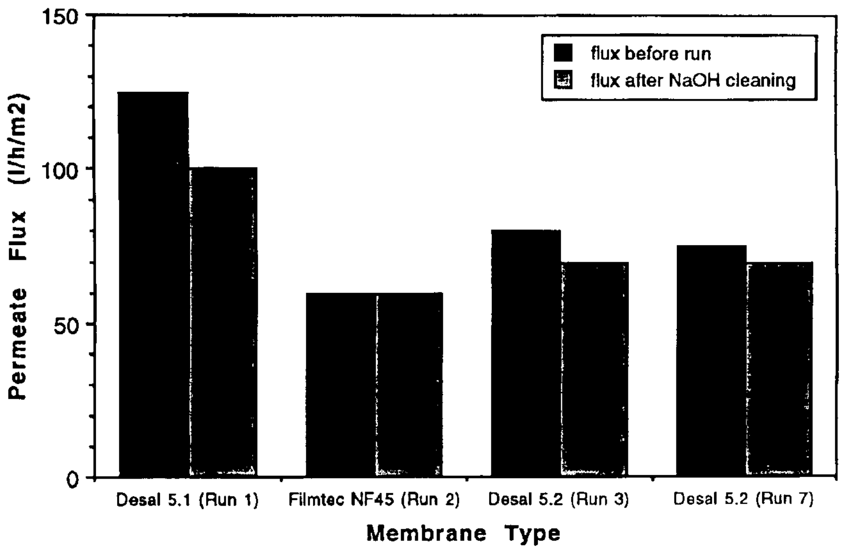

Fig. 8. Effect of effluent contact on flux performances of various membranes used with a 20 g/l NaCl solution at 15 bar.

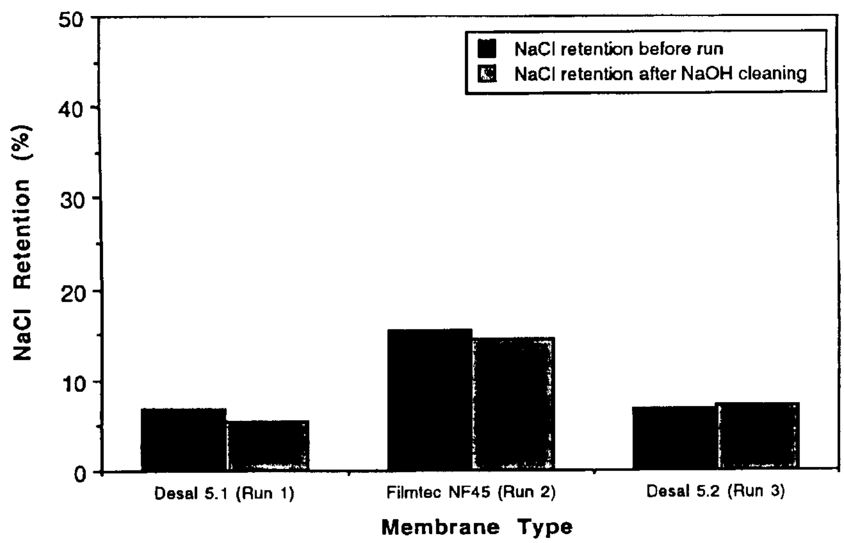

Fig. 9. Effect of effluent contact on sodium chloride retention of various membranes used with a 20 g/l NaCl solution at 15 bar.

experiment. However, the next results obtained in Run 8 with Membrane Desal 5.1 were similar to those with Membrane Desal 5.2. Thus, the initial membrane deterioration may be due solely to the manufacturing process.

8. Conclusions and perspectives

The organic spiral-wound NF membranes tested in this study demonstrated satisfactory qualitative performances for the recovery of sodium chloride and the removal of organics matter from the regeneration waste in the permeate. The NF process allowed the achievement of a 74% reduction in salt consumption and an 89% reduction in water consumption while reducing the volume of toxic waste discharged from sugar refineries.

Decline in the permeate flux rate due to membrane fouling was observed in both total recycle and batch concentration experiments. However, the exposure of membranes to

regeneration effluent did not induce any membrane alteration.

While the results obtained in this study are promising, further research is needed to compare the efficiency of brine permeate and fresh brine to regenerate loaded ion-exchange resins.

Additional development work is still needed for secondary treatment of highly colored retentate generated by NF such as precipitation with lime and phosphoric acid in order to make the overall process attractive to other sugar refineries.

9. Symbols

J_p	—	Permeate flux, l/h/m^2
[NaCl]	—	Sodium chloride, g/l
OD	—	Optical density of feed
Rec_{NaCl}	—	Sodium chloride recovery in permeate, %
Rem_{Col}	—	Colorant removal in permeate, %
Ret_{Col}	—	Colorant retention, %
Ret_{NaCl}	—	Sodium chloride retention, %
VCF	—	Volumetric reduction factor
V	—	Volume of feed, l

Subscripts

f	—	Feed
p	—	Permeate
r	—	Retentate

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